VAMAS intercomparison of critical current measurements on Nb_3Sn superconductors: a summary report

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This paper is a summary of an international collaboration endorsed by VAMAS to study problems associated with critical current measurements in Nb_3Sn superconductors and provide guidelines for a standard measurement. Two series of critical current measurements were implemented. In the first series, three different sample conductors were used and participants made measurements using their own techniques. As a result, coefficients of variation for these samples at 12 T turned out to be 8–29.9%. A major source of these variations was attributed to strain sensitivity of the Nb_3Sn conductors. Thus, the second series of measurements were done on one sample conductor and under specified measurement conditions, particularly in terms of specimen strain. The coefficient of variation decreased to 2.2%, which is regarded as a reasonable base for future establishment of an international standard measurement method.

Keywords: Nb_3Sn; critical current measurement methods; interlaboratory comparison; standardization

To complement the rapid, worldwide progress in superconductivity related science and technology, collaboration has occurred between workers in Japan, Europe and the USA to move towards standardization of data on superconducting materials. The importance of standardization was recognized at the Economic Summit of Heads of State held at Versailles in 1982. An international technical working party (TWP) was formed within the framework of VAMAS (Versailles Project on Advanced Materials and Standards). Since its formation in 1986 the TWP has been working on the standardization of critical current measurements for Nb_3Sn and a.c. loss measurements for NbTi multifilamentary composite superconductors. This paper summarizes the work on Nb_3Sn, which was based on comparative data gathered in two consecutive programmes involving more than 30 laboratories. A full description will be published in the near future in a Cryogenics supplement[1].

First intercomparison of critical current measurements

Table 1 is a list of 25 laboratories which participated in the first intercomparison programme. The programme was established partly by learning from preliminary efforts which had been made towards standardization[2–5].

Test procedures

1. Organization

Three central laboratories organized sample preparation, distribution and heat treatment of the test samples. These laboratories were the National Research Institute for Metals (NRIM), Japan, the Bureau Central de Mesures Nucleaires (BCMN), Europe, and the National Institute of Standards and Technology (NIST), USA. NRIM collected all the results for preliminary analysis, which were then presented at TWP meetings for discussion. Some of the results have been published elsewhere[6–8].

2. Sample conductors

Relatively low current capacity conductors were chosen (<500 A at 7 T) so as not to exclude possible participants with limited power supplies. Three essentially different conductors, labelled as samples A, B and C, were included in the programme. The specifications of these samples are given in *Table 2*.

3. Reaction heat treatments

To assess the effect of possible variations in the reaction heat treatments at different laboratories, each participant

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Table 1 List of participant laboratories in the first intercomparison

Japan	Electrotechnical Laboratory Furukawa Electric Hitachi Japan Atomic Energy Research Institute Kobe Steel National Research Institute for Metals Osaka University Tohoku University
Europe	Atominstitut der Oesterreichschen Unv (Austria) Bureau Central de Mesures Nucleaires (Belgium) Clarendon Laboratory (UK) CNRS/SNCI (France) ENEA Centro di Frascati (Italy) Kernforschungszentrum Karlsruhe (Germany) Nijmegen University (Netherlands) Rutherford Appleton Laboratory (UK) SCK/CEN (Belgium) Siemens (Germany) Technische Universitaet Wien (Austria) Vacuumschmelze (Germany)
USA	Brookhaven National Laboratory Lawrence Livermore National Laboratory Massachusetts Institute of Technology National Institute of Standards and Technology University of Wisconsin

was supplied by a central laboratory with enough wire from each sample type to make two specimens. The participant divided the supplied wires into two pieces and wound them on to reaction mandrels of his own design and fabrication. After mounting, one of each sample type was returned to the central laboratory for 'central reaction', while the other was retained for 'self reaction' by the participant.

In most cases, the reaction mandrels were stainless steel tubes with surface treatments designed to avoid diffusion bonding between the specimen and the mandrel during heat treatment. Some laboratories used the same mandrel for reaction and measurement to reduce the possibility of damage to the specimen due to handling, since variations due to such damage cannot easily be distinguished from those due to variations in heat treatment.

Some heat treatments were carried out in argon or hydrogen, although most were done in a vacuum. Each sample was reacted according to manufacturer's directions. These were 700°C for 96 h, 670°C for 200 h and 700°C for 48 h for samples A, B and C, respectively.

4. Measurement guidelines

A four terminal resistance measurement was adopted and participants were requested to measure the critical current at electric fields of 5, 10 and 100 μV m^{-1} and at integer magnetic fields. Resistive criteria of 10^{-14} and 10^{-13} Ωm were also acceptable. Participants were requested to determine the value of n for the empirical equation

$$V \propto I^n \tag{1}$$

in which V and I are the voltage and current across a sample specimen, respectively.

Participants were encouraged to use their own measurement techniques so that sources of measurement variability could be identified. Details of the specimen mounting technique, the measurement system and the experimental conditions adopted at each laboratory were reported using standardized data sheets.

5. Survey of measurement parameters

Sample mounting. In each laboratory the specimen was wound on to the mandrel in an open spiral. The mandrel was attached to a probe which was subsequently inserted into the magnet. A retaining groove was often made on the mandrel to hold the specimen in place.

Mandrels were made from stainless steel, hastelloy, brass and copper, to which the specimen was often soldered. Fibreglass and ceramics were also used, and in these cases the specimen was often affixed using grease or resin. Some measurements were carried out with no bonding agent.

Copper rings were attached to each end of the mandrels to provide current terminations, and voltage taps were soldered directly to the specimen. The distance between the copper rings and between the voltage taps varied considerably. The shortest specimen length was 135 mm which required 1.5 turns on the mandrel, while the longest specimen was 2000 mm long, requiring 30 turns.

Depending on the direction of current flow, the coiled specimen can experience radially inward or outward Lorentz forces. An outwardly directed force results in a tensile hoop stress, whereas an inwardly directed force is transmitted directly to the mandrel. Only two laboratories opted for an outward Lorentz force. Some participants investigated the effect of current direction on the measurements.

Magnetic and electric parameters. Participants used superconducting and combinations of superconducting and copper solenoids to generate magnetic fields typically up to 16.5 T and in some cases over 20 T. The magnetic field was measured using NMR, a Hall probe or a rotating magnetometer. The accuracy of the magnetic field measurement and a survey of other measurement parameters are shown in *Figure 1*.

Table 2 Specifications of samples used

	Sample A	Sample B	Sample C	NbTi
Fabrication method	Bronze	Bronze	Internal tin	
Wire diameter (mm)	0.8	1.0	0.68	0.51
Structure	NbTa/CuSn	Nb/CuSnTi	Sn/Cu/Nb	NbTi/Cu
Cu to non-Cu ratio	0.22	1.68	0.88	1.8
Bronze/filaments ratio	2.8	2.5	3.1	
Filament diameter (μm)	3.6	4.5	2.7	23
Number of filaments	6156	5047	5550	180

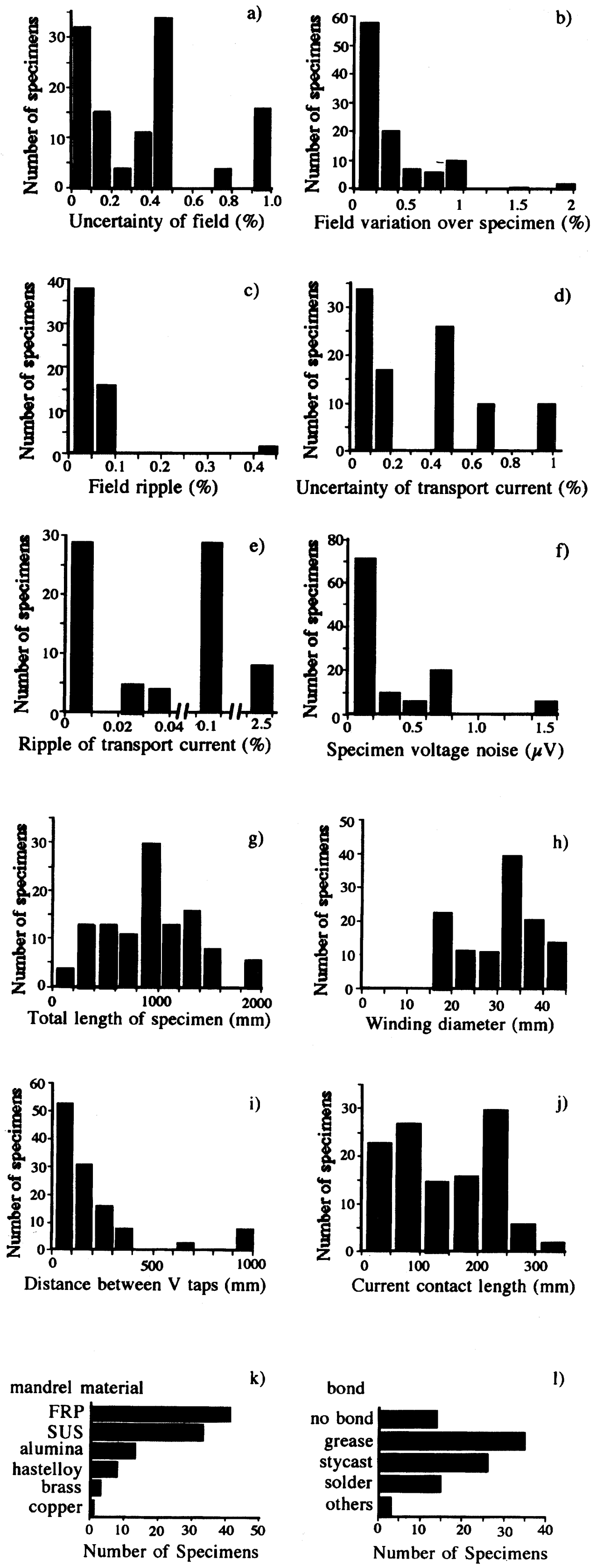

Figure 1 First intercomparison frequency distributions with respect to: (a) uncertainty of applied field; (b) field variation over specimen; (c) field ripple; (d) uncertainty of transport current; (e) ripple of transport current; (f) specimen voltage noise; (g) total length of specimen; (h) winding diameter; (i) distance between voltage taps; (j) current contact length; (k) measurement mandrel material; and (l) bond used to fix specimen to measurement mandrel

Results and discussion

Critical current measurements were made on central and self reacted specimens of samples A, B and C, resulting in six data sets. *Figure 2* is a log/linear plot of critical current as a function of magnetic field at an electric field criterion of 10 μV m^{-1} for sample B (central reaction). Although there were different variations in magnitude, the plot is typical of the five other data sets. The data fit the Kramer equation[9] in the range from less than 6 T to at least 12 T with, in many cases, an accuracy of better than $\pm$0.5%. Two key factors revealed by fitting to the Kramer equation are: 1, a systematic and inexplicable variation is consistently observed in the data from some laboratories; and 2, random variations in individual data points are clearly visible and indicate the accuracy with which individual data have been obtained[10].

The coefficient of variation (defined as the ratio of the standard deviation to the average value at any field) is generally not constant as the magnetic field is changed. *Table 3* contains a statistical summary of critical current measurements made for samples A, B and C under central and self reaction at various magnetic fields. Two rows of the table show a reduction in the coefficient of variation with increasing magnetic field, in spite of the fact that field measurement errors, strain variation and temperature variation should all act to increase the coefficient of variation as the field is increased. One possible reason for this observed reduction may be the inclusion of non-overlapping data. Sample C was the only internal tin conductor and showed the highest coefficients of variation among the three sample conductors. This seems to be due to its high sensitivity to strain, as discussed below.

The critical current was measured under conditions where the self-field was parallel and anti-parallel to the background field. The major effect was expected to be due to strain in the superconducting filaments, which is reduced when the composite is stretched. The measured values were the same for both cases when the specimen was strongly bonded to the measurement mandrel. However, when the

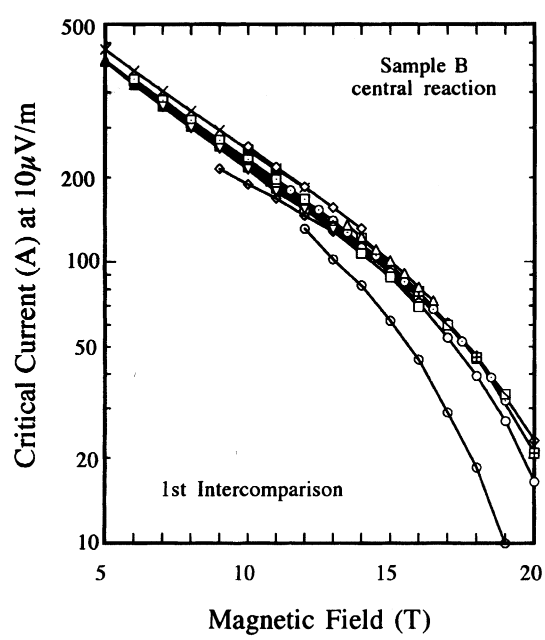

Figure 2 I_c(10 μV m^{-1}) *versus* magnetic field curves for centrally reacted sample B in the first intercomparison

Table 3 Interlaboratory averages and standard deviations of I_c at 10 μV m^{-1} in the first intercomparison

	Magnetic field (T)	8	10	12	14
Sample A, central reaction	Number of data	5	9	11	10
	Average of I_c(A)	366.94	268.59	188.24	137.91
	S.D. (A)	61.08	41.02	33.04	24.97
	S.D./average (%)	16.6	15.3	17.6	18.1
Sample A, self reaction	Number of data	9	12	10	5
	Average of I_c(A)	371.12	265.06	188.62	125.44
	S.D. (A)	12.05	16.98	18.27	12.96
	S.D./average (%)	3.2	6.4	9.7	10.3
Sample B, central reaction	Number of data	9	17	18	17
	Average of I_c(A)	313.77	225.89	162.22	112.91
	S.D. (A)	12.65	15.67	13.02	10.92
	S.D./average (%)	4.0	6.9	8.0	9.2
Sample B, self reaction	Number of data	8	13	11	8
	Average of I_c(A)	362.12	246.92	157.27	107.99
	S.D. (A)	60.83	36.68	14.55	12.01
	S.D./average (%)	16.8	14.9	9.3	11.1
Sample C, central reaction	Number of data	9	15	18	14
	Average of I_c(A)	209.60	127.85	67.09	28.26
	S.D. (A)	57.66	41.47	20.09	9.14
	S.D./average (%)	27.5	32.4	29.9	32.3
Sample C, self reaction	Number of data	7	11	7	5
	Average of I_c(A)	204.81	147.14	84.23	36.80
	S.D. (A)	54.60	28.35	12.55	55.34
	S.D./average (%)	26.7	19.3	14.9	14.5

specimen was bonded with grease, slightly larger critical currents were often obtained when the fields were parallel.

The following strain related observations were made in addition to those noted above:

(i) The measured critical currents of specimens mounted and soldered on to stainless steel, brass or copper mandrels were somewhat smaller than average values, due to the greater contraction of metals compared to fibreglass reinforced plastic (FRP) or ceramic. The critical current variability was also small, in contrast to the higher variability of specimens mounted and glued to FRP mandrels. This probably arose from the use of mandrels with different wall thicknesses and different types of FRP. A lower variability was observed when the specimen was weakly bonded or not bonded at all to the mandrel.

(ii) The measured critical currents of specimens mounted on ceramic mandrels tended to be above the average values. This is probably because of the lower contraction of ceramic which, on cool-down, results in tension in specimens. This, in turn, relieves the precompression in the superconducting filaments.

Complementary studies

1. Homogeneity of sample conductors

The longitudinal I_c homogeneity of samples A and B was assessed using 21 specimens from sample A and seven from sample B. The specimens were taken at intervals along the entire lengths of the samples and wound on reaction mandrels. Heat treatment of the specimens was carried out at the Culham Laboratory under contract to the Rutherford Appleton Laboratory (RAL). No assessment of homogeneity was carried out on sample C.

Critical current measurements on the 21 specimens from sample A were made at SCK/CEN at a temperature of 4.3 K and magnetic fields of 7–10 T. The coefficients of variation are 1.5% at 7 T and 2.4% at 10 T.

The homogeneity of sample B was examined at the Clarendon Laboratory. Critical current measurements were carried out at 4.2 K and 7–15 T. Standard deviations were within 1.3% at all magnetic fields and electric criteria studied.

2. Measurements of I_c at different levels of specimen strain

These measurements were carried out at Osaka University and at NIST on short straight specimens (30–50 mm) and at RAL, where the specimen (>700 mm) was mounted on a strain spring[11] in a geometry similar to a conventional short specimen. *Figure 3* shows the normalized critical current as a function of strain for samples A, B and C for short straight specimens. This plot indicates a parabolic curve with a peak at zero strain in the superconducting filaments. The strain spring results indicated a linear relationship on either side of the peak. The magnitude of the variations with strain was similar in all three series of measurements.

Figure 3 shows that sample C is more sensitive to strain at 15 T compared to the other samples. This is primarily due to its relatively low critical field of 19 T compared to 24.5 T for the other two samples. The difference between samples A and B cannot be as easily accounted for; it seems likely to be due to structural differences within the conductors.

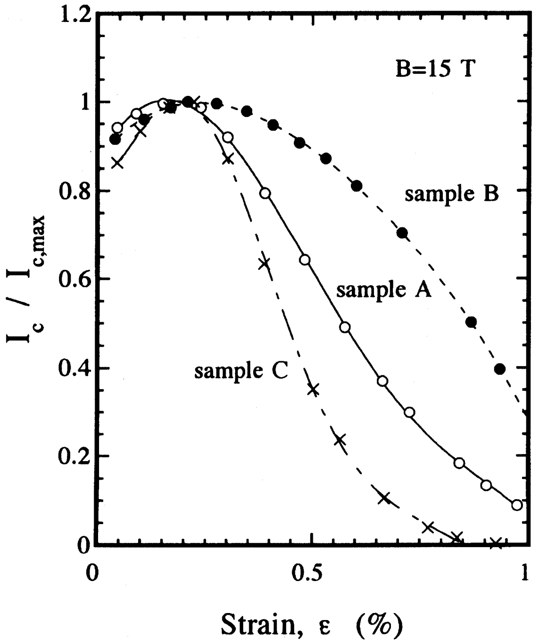

Figure 3 Strain effect measurement carried out, comparing three test samples in the first intercomparison

3. Thickness effects arising from use of FRP mandrels

In a series of measurements at NIST using specimens from samples A, B and C, it was observed that the critical current was higher for specimens mounted on thin walled mandrels compared to thick walled mandrels. In fact, due to the anisotropic structure of FRP, thick walled mandrels contract more on cool-down to 4.2 K than thin walled mandrels. Thus the strain in a specimen depends on the wall thickness, which in turn affects the critical current.

Summary of the first intercomparison of critical current measurements

(i) The homogeneity study showed that the samples had high longitudinal homogeneity.
(ii) The coefficients of variation of critical current at magnetic fields between 8 and 14 T were 15–18%, 4–9% and 27–33% for centrally reacted samples A, B and C, respectively. Sample C had the highest coefficients of variation because it was most sensitive to strain and reaction heat treatment conditions. The coefficients of variation generally increased with increasing magnetic field.
(iii) Reaction and measurement mandrel materials had a significant effect on the critical current. There are advantages and disadvantages for using each material.
(iv) The specimen mounting method and the tightness of the mounting significantly affected the measured critical current.
(v) n-Values were small when the specimen was soldered on to a metallic mandrel.
(vi) The specimen strain can be a significant source of critical current measurement variability.

Second intercomparison of critical current measurements

The variations seen in the first intercomparison were larger than would be acceptable from a standard measurement method. Thus, a second intercomparison, in which measurement techniques would be more closely specified, was proposed at a meeting in Karlsruhe in 1990. As listed in *Table 4*, 13 laboratories from six countries participated in the measurements.

Evolution of the first to second intercomparison

In the first intercomparison, variations in the measured critical current due to variations in the sample and heat treatment were anticipated. However, to avoid prejudging any technique, almost no restrictions were placed on the measurement methods. Instead, participants were encouraged to use such methods as were developed by each of them individually over many years. It is notable therefore that all participants coiled and mounted the specimen on to a cylindrical mandrel with current terminations attached to its ends. The specimen length was typically more than six turns around the mandrel. In all cases, voltage was measured over a central section remote from the current terminations. The only clear differences were the materials from which the mandrel was made and the bonding agent used. The choice for these materials resulted from individual experience of the conflicting advantages and disadvantages.

An attempt was made to incorporate the experience from the first intercomparison into the design of the second intercomparison. Since specimen strain due to the thermal contraction of the measurement mandrel can significantly affect conductor performance, it was decided that, in addition to specification of techniques and measurements, the measurement mandrels would be made to a common specification.

For the second intercomparison, specimens from a single Nb_3Sn conductor were measured. In addition, measurements were made on specimens of a standard NbTi conductor. Since NbTi conductors are largely insensitive to mechanical effects, variation in measurements on them would indicate the level of variation in calibrations or environment at the participating centres.

Complementary measurements on the variation of current with field and a further independent variable were performed as follows on the chosen Nb_3Sn sample.

Table 4 List of participant laboratories in the second intercomparison

Japan	Electrotechnical Laboratory Hitachi Tohoku University Kobe Steel National Research Institute for Metals
Europe	Atominstitut der Oesterreichschen Unv (Austria) CNRS/SNCI (France) ENEA Centro di Frascati (Italy) Kernforschungszentrum Karlsruhe (Germany) Technische Universitaet Wien (Austria) Vacuumschmelze (Germany)
USA	National Institute of Standards and Technology Teledyne Wah Chang Albany

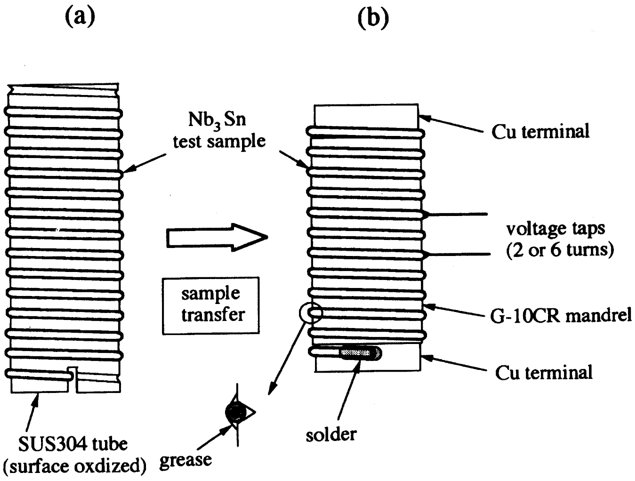

Figure 4 Schematic illustration of (a) reaction mandrel and (b) measurement mandrel on which Nb_3Sn sample specimen is mounted in the second intercomparison

Test procedures

1. Organization

NRIM was designated as the central laboratory for the second intercomparison and was responsible for the reaction and supply of specimens from the Nb_3Sn sample conductor. It was also responsible for supply of the NbTi samples and for the materials from which the measurement mandrels were made. The individual participants constructed the measurement mandrels, transferred the specimens from the reaction mandrels on which they were supplied and measured the critical current.

2. Sample conductors

The sample B conductor used in the first intercomparison was also used as the Nb_3Sn sample in the second intercomparison. The NbTi sample was the NIST standard reference material SRM-1457[12], specifications for which are given in *Table 2*.

3. Reaction heat treatment

Identical cylindrical stainless steel reaction mandrels were produced, each with a spiral groove machined into its cylindrical surface, as illustrated in *Figure 4a*. They were subsequently oxidized by heating for 3 h in air at 800°C in order to prevent diffusion bodning to the specimen during reaction. The specification for these mandrels is given in *Table 5*. The sample conductor was cut into lengths of ≈ 1.0 m. Each length was wound into the groove on one of the mandrels. Reaction heat treatment of 670°C for 200 h in a dynamic vacuum of 4×10^{-5} torr* was finally carried out with all the specimens in one batch. The temperature was controlled to within ±1.0°C in time and space.

Table 5 Specifications of reaction and measurement mandrels in the second intercomparison

	Reaction mandrel	Measurement mandrel
Material	304 stainless steel	G-10CR plate tube
Outer diameter (mm)	24.93	25.00
Inner diameter (mm)	~19	Not specified
Groove shape	60° 'V'	60° 'V'
Groove pitch (mm)	3.0	3.0
Spiral turns	13	10

4. Measurement mandrel and specimen mounting

Any one of the original mandrel designs might have been chosen. For example, the low thermal contraction on cool-down of ceramic mandrels results in the least stress in the superconducting filaments and would therefore have resulted in the highest values of critical current; while specimens soldered to metallic mandrels would have relatively small variations in measured critical current due to differential thermal contraction.

However, a design using G-10CR (FRP) was chosen because its contraction on cool-down was close to that of the overall contraction of the particular Nb_3Sn composite superconductor used in the programme. This was considered to be a desirable feature for a standard mandrel. Also, since the mandrel material is non-metallic, current does not bypass through the mandrel. The CR grade of G10 was chosen because its higher manufacturing specifications result in better performance at low temperatures.

Cubes of material of 5 cm^3 were supplied. From these, each participant was required to machine a plate tube in which the fibreglass fabric planes were perpendicular to the axis, the direction in which a thermal contraction match with the specimen is obtained. The specifications are found in *Table 5*. *Figure 4b* shows a 3 mm pitch spiral groove which matches that in the reaction mandrels. Copper current connection rings were then added to each end to complete the mandrel, as shown in *Figure 4b*.

The specimens were required to be transferred with great care and minimum deformation from the reaction to the measurement mandrels. They were also to be completely coated in silicon grease. One end of the specimen was to be soldered to a current connection ring and, starting from that end, the specimen was to be progressively pressed into the groove, to avoid slack, until the other end was reached. This end was then to be soldered to the other connection ring. Grease, which solidifies on cool-down, was the chosen bonding agent because the specimen can later be removed from the mandrel. Varying thicknesses of grease were later reported by the participants and, contrary to requirement, some of them applied it after soldering the specimen.

The NbTi samples were more robust but were required to be carefully treated while they were being attached to the mandrels as prescribed for the Nb_3Sn sample. The insulation on the NbTi sample was removed chemically to allow the sample to be soldered.

Two pairs of voltage taps, each centred on the midpoint, were to be soldered to the specimens; the first pair to be separated by two turns and the second by six turns. It was subsequently found, with the Nb_3Sn specimens, that the difference between measured critical currents based on the voltage generated over two or six turns was less than 1.0%, and was less than 10% for the *n*-value. This indicated that end effects would be absent at the inner pair of taps and

* 1 torr = 133.322 N m^{-2}

subsequent analysis could therefore confidently be based on measurements associated with them.

5. Measurement requirements and conditions

Participants were required to determine the critical current of the Nb_3Sn sample at 10 and 100 $\mu V\ m^{-1}$ and at magnetic fields of 8, 9, 10, 11 and 12 T. For the NbTi sample the field levels were to be 6, 7, 8 and 9 T. Values of n in Equation (1) were required to be derived from at least four data points on the *V–I* curve at all field levels, using the least squares method.

Furthermore, it was required to cool down the specimens uniformly from room temperature to liquid helium temperature over a period of at least 5 min. This was fulfilled by all participants. It was also required that the specimen current be orientated to produce a radially inward directed Lorentz force. This was also adhered to by all participants.

Critical current measurements were required to be started from the magnetic field at which the Lorentz force is greatest, with a last repeat measurement to be again taken at this field. This data can be used to determine the change in the specimen that may have occurred during the series of measurements. However, some participants started from their highest available field and carried on downwards. In most cases the difference between first and last measurements was less than 1.0%.

Results

1. Comparison of critical currents obtained on Nb_3Sn sample

Figure 5 is a log/linear plot of critical current as a function of magnetic field. A comparison with *Figure 2* clearly shows the reduction in critical current measurement variability.

Averages and standard deviations are listed in *Table 6*. Average values of critical current at 12 T obtained at 10 and 100 $\mu V\ m^{-1}$ were about 2% higher than those in the

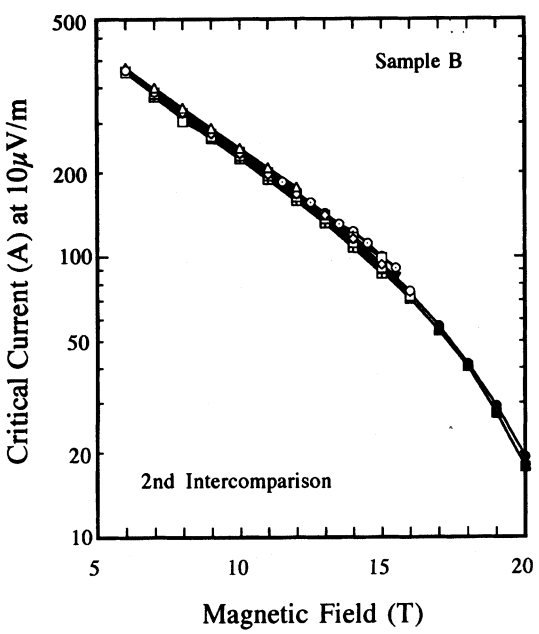

Figure 5 $I_c(10\ \mu V\ m^{-1})$ *versus* magnetic field curves for Nb_3Sn sample in the second intercomparison

Table 6 Interlaboratory averages and standard deviations of $I_c(10\ \mu V\ m^{-1})$ for Nb_3Sn sample in the second intercomparison

Magnetic field (T)	8	10	12	14
Number of data	14	14	19	15
Average of I_c(A)	322.21	233.17	165.68	114.56
SD (A)	7.13	4.23	3.58	3.69
SD/average (%)	2.2	1.8	2.2	3.2

first intercomparison, and average values of n were 10% higher. However, the coefficients of variation were greatly reduced from 8.0, 8.1 and 20.6% in the first intercomparison to 2.2, 2.5 and 7.1% in the second intercomparison for critical currents at 10 and 100 $\mu V\ m^{-1}$, and n, respectively.

Eight participants made identical measurements on two specimens of the Nb_3Sn sample conductor. Three of the participants found differences of less than 1% between their two specimens. However, in contrast to the reduction in variation, differences of up to 5% between the specimen pairs were found by the other five participants. The reason for this may be the variation in sample homogeneity and non-uniform experimental variables such as strain or sample transfer from reaction to measurement mandrel.

2. Comparison of critical currents obtained on NbTi sample

Averages and standard deviations are listed in *Table 7*. At 10 $\mu V\ m^{-1}$ and 6 and 8 T the values are almost identical to the NIST certified values of 122.6 and 68.08 A with total uncertainties of 1.71 and 1.97% , and coefficients of variation of 0.36 and 0.35%, respectively[2,13].

From these results it may be concluded that the specified measurement procedures are well suited to critical current measurements in NbTi, and that the variation due to sample inhomogeneity is less than one-third and one-sixth of the observed variation at 6 and 8 T, respectively.

Complementary studies

1. Effects of temperature variation

The effects of temperature variation were examined at the Technische Universitaet Wien (TUW) and NIST. The rate of change of current with temperature at 4.2 and 2.4 K in the Nb_3Sn sample was linear, with typical values for dI_c/dT of 30–33, 26–28 and 21–24 A K^{-1}, at magnetic field levels of 8, 10 and 12 T, respectively.

2. Strain effects

Strain effects were examined at NIST. Using the Nb_3Sn specimens included with the original batch for heat treatment at NRIM, the strain at maximum current was

Table 7 Interlaboratory averages and standard deviations of $I_c(10\ \mu V\ m^{-1})$ for NbTi sample in the second intercomparison

Magnetic field (T)	6	7	8	9
Number of data	12	12	12	12
Average of I_c(A)	122.87	95.43	68.28	41.00
SD (A)	1.40	1.38	1.43	1.51
SD/average (%)	1.1	1.45	2.1	3.7

$0.25 \pm 0.02\%$ and the maximum strain below which the effect of strain is reversible was $0.92 \pm 0.04\%$. A good fit was also found to the Ekin's strain scaling law[13].

3. Effects of mandrel materials

The effects of mandrel materials were examined at TUW and Teledyne Wah Chang Albany (TWCA) using stainless steel mandrels in addition to the common FRP (G-10CR) mandrels. Due to differential thermal contraction, the Nb_3Sn specimen bonded to the stainless steel mandrel should have a smaller critical current than when bonded to the G-10CR mandrel. One laboratory reported a substantially smaller critical current with the stainless steel mandrel compared to the G-10CR mandrel, as expected. However, the other reported a larger value. This difference may be interpreted by the possible difference, for example, in thickness of grease layer covering the specimen. This thickness should affect thermal and mechanical conditions around the specimen.

4. Effects of mandrel dimensions

The effects of mandrel diameters and groove pitches were examined at Kobe Steel. Critical currents of Nb_3Sn specimens bonded with silicon grease to G-10CR mandrels showed some variation with mandrel outer diameters (25 and 35 mm), corresponding to the field profile within the magnet used. Effects of mandrel wall thickness and groove pitch were not appreciable.

5. Effects of specimen bonding materials

Effects of specimen bonding materials were examined at TUW. There was little difference between grease and Stycast when used on G-10CR or stainless steel mandrels. It was found, however, that the absence of any bonding material resulted in a reduction in the measured critical current. It is possible that a small radial clearance was present between specimen and mandrel after winding, and this was taken up by compression of the specimen as the Lorentz force was created. The result of this would be an increase in the compressive strain in the superconducting filaments and a corresponding reduction in the critical current, as observed.

Discussion

The variations observed in the second intercomparison were significantly less than those observed in the first. It is reasonable to assume that the difference was due to the control exercised over the measuring conditions in the second intercomparison, as detailed in *Table 8*. The measurement guidelines should take the origins of the variability into consideration.

1. Origins of variability in critical current measurement on NbTi

Measurements on the NbTi conductor were intended to separate out effects of variation in strain from those due to variation in all the other parameters, since the superconducting properties of NbTi are insensitive to strain. Critical current homogeneity of the long range (among spools) and the short range (within a spool) has been estimated in terms of the coefficient of variation to be 0.22% and 0.27% at 6 T, respectively[2]. These values are substantially smaller compared to the total coefficients of variation found on the NbTi sample in the second intercomparison (1.1–2.1% for fields between 6 and 8 T; see *Table 7*). Thus, the effect of critical current homogeneity variation on the variability of critical current data in the NbTi sample is considered to be small.

The reported accuracy of applied field determination (central field and field profile) varies between 0.2 and 2%. If the actual field is higher than the assumed value by 1%, critical current would be reduced by 1.3 and 3.2% at 6 and 8 T, respectively[12]. The deviation in field at each laboratory was estimated and found at NRIM[1] to correspond to 0.8 and 2.1% variations in critical current at 6 and 8 T, respectively. As described above, the total variation coefficients found on the NbTi sample in the second intercomparison are 1.1 to 2.1% for fields of 6 and 8 T, respectively. Thus, more than 50% of the total data scatters on the NbTi may be attributed to applied field variation. In other words, the accuracy in applied field determination may considerably affect the magnitude of critical current data scatter.

2. Origins of variability in critical current measurement on Nb_3Sn

In the first intercomparison, a study of critical current homogeneity on the Nb_3Sn sample performed at the Clarendon Laboratory showed that the coefficient of variation for seven specimens taken from one long sample B conductor was $\approx$1.3% at 12 T.

Most of the reported liquid helium bath temperatures, with respect to the Nb_3Sn sample, are 4.219 ± 0.023 K. If the temperature is raised by 0.02 K, critical current will be decreased by 0.3% at 12 T. Thus, the variation in measuring temperature among participant laboratories is not expected to be a major source of variability.

Applied fields were corrected for critical current measurements on the Nb_3Sn sample. The critical currents (electric field criterion of 10 $\mu V\ m^{-1}$) for Nb_3Sn at 12 T decreased with increasing corrected field, suggesting that over- and underestimations on critical current for 12 T occurred due to the applied field deviations from 12 T at different laboratories. The accuracy of applied field determination is estimated to be ±0.6% at 12 T, which corresponds to a coefficient of variation of 1.4%. This suggests that the deviation in a field has an essential contribution to the critical current data variability.

The strain effect on the Nb_3Sn sample can be described using Ekin's strain scaling law[13]. According to the fitting of the strain effect measurement data at 12 T with the strain scaling law, a ±0.03% in strain could result in a coefficient of variation of 1.3%. It should be noted that a strain of ±0.03% is close to the limit of control for a critical current measurement.

3. Evaluation of sources of variability

Contributions of different measurement variables to the variability in critical current for the Nb_3Sn sample were estimated and are listed in *Table 9*. Contributions from variations in specimen current and voltage were estimated using the averages of reported measurement accuracies. *Table 6* shows that the magnitude of total critical current variation for 10 $\mu V\ m^{-1}$ and at 12 T in terms of coefficient

Table 8 Comparison of the first and second intercomparisons of critical current measurements

	First intercomparison	Second intercomparison
Sample conductor	3 Nb_3Sn wires	1 Nb_3Sn wire
Heat treatment site	3 central labs and each participant lab	1 central lab
Reference material	None	NbTi wire: NIST SRM-1457
Sample configuration	Spiral: dimensions and geometry not specified	Spiral: diameter 25 mm; pitch 3 mm; turns 12
Voltage tap location on specimen	Not specified	Specified
Mandrel material	Not specified	Specified: FRP (G-10CR)
Specimen mounting	Not specified	Specified: silicone grease
Specimen temperature	Not monitored	Monitored
Applied field	Not calibrated	Calibrated
Complementary studies	I_c homogeneity, strain effect	Strain effect, mandrel dimensions, mounting methods, I_c *versus* temperature

Table 9 Contributions of measurement variables to variability in I_c for Nb_3Sn in the second intercomparison

Measurement variables	Variations (mean square root) (%)	Variability in I_c at 12 T (%)
Sample current	±0.4	±0.4
Sample voltage	±5.0	±0.2
Temperature	±0.6	±0.4
Magnetic field	±0.6	±1.4
Strain	<±0.03	<±1.3
I_c homogeneity		±1.0

of variation fell from 8.0% in the first to 2.2% in the second intercomparison. Assuming that the contributions from factors other than strain were the same in both intercomparisons, the measurement requirements in the second intercomparison, which were intended to limit the influence of strain, have reduced the data variability by as much as 6.4%. If the rest of the variability, 1.3%, is attributed to uncontrolled strain, it corresponds to a strain variation less than 0.03%. On the other hand, it was shown in both NbTi and Nb_3Sn measurements that applied field deviation can significantly affect the critical current data scatter.

Based on the two intercomparisons, it is assumed that the variability in measured critical current in the Nb_3Sn composite superconductor originates mainly from variations in specimen strain and applied field.

Summary of second intercomparison

Results of the second intercomparison may be summarized as follows.

(i) The variability of critical current for the Nb_3Sn sample is comparable with that for the NbTi reference sample.

(ii) In the second intercomparison, the coefficients of variation for critical current ($10\,\mu V\,m^{-1}$) and *n*-value on the Nb_3Sn sample at 12 T were 2.2 and 7.1%, respectively. In the first intercomparison, these values were 8.0 and 20.6%.

(iii) The major sources for the critical current data scatter may be identified as variations in specimen strain and applied field. This indicates that specimen handling must be specified precisely.

Conclusions

Two series of interlaboratory comparisons of critical current measurements were implemented with the participation of more than 30 laboratories from Japan, Europe and the USA. The results were discussed by the technical working party (TWP) formed by researchers from the participating laboratories. Based on these intercomparisons, together with complementary studies, it may now be concluded that the measurement requirements in the second intercomparison were, as a whole, effective for reducing the critical current data scatter on the examined Nb_3Sn sample conductor. In particular, those requirements that specify sample handling or sample strain conditions are important. By adopting these requirements, the variability in critical current data was reduced to 2.2%, which should be regarded as a meaningful accomplishment for the evaluation of Nb_3Sn conductors for regular superconducting magnet use. Thus, these requirements can provide a basis for establishing future standard critical current measurement guidelines.

For a higher level of measurement accuracy or superconductors for special uses, however, more detailed studies may be needed in which mandrels and specimen mounting methods are more extensively examined and applied field is more precisely calibrated. In addition to the work described in this paper, the TWP has defined a superconducting materials terminology, to provide engineers in the field of superconductivity related technologies with a common base of understanding of superconducting materials and some areas of their application. This appears as an appendix in a forthcoming supplement[1]. It should also be mentioned that interaction with an international standardization organization, the International Electrotechnical Commission (IEC), has already occurred, whereby experience obtained through the VMAS efforts is regarded as significant to the progress in the international standardization of superconducting materials.

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References

1 **Wada, H., Goodrich, L.F., Walters, C.R. and Tachikawa, K. (Eds)** The VAMAS Technical Working Area 6: Superconducting and Cryogenic Materials Report. Critical current measurement method for Nb_3Sn multifilamentary composite superconductors, manuscript in preparation to appear in a *Cryogenics* supplement

2 **Goodrich, L.F., Vecchia, D.F., Pittman, E.S., Ekin, J.W. *et al.*** Critical current measurements on an NbTi superconducting wire standard reference material, NBS Special Publication 260-91, National Bureau of Standards, Boulder, CO, USA (Sept 1984)

3 **Ekin, J.W.** Preliminary results on US/Japan round robin II *Proc 5th US-Japan Workshop on High-Field Superconductors* Fukuoka, Japan (1987) 168

4 **Nagata, A. Watanabe, K. and Noto, K.** Results of I_c round robin test in Japanese universities *Proc 5th US-Japan Workshop on High-Field Superconductors* Fukuoka, Japan (1987) 188

5 **Itoh, K.** Critical current round robin test – results at NRIM and other Japanese laboratories *Proc 5th US-Japan Workshop on High-Field Superconductors* Fukuoka, Japan (1987) 196

6 **Tachikawa, K.** VAMAS intercomparison of critical current measurements in superconducting Nb_3Sn wires *Cryogenics* (1989) **29** 710

7 **Tachikawa, K., Itoh, K., Wada, H., Gould, D. *et al.*** VAMAS intercomparison of critical current measurement in Nb_3Sn wires *IEEE Trans Magn* (1989) **MAG-25** 2368

8 **Wada, H. and Itoh, K.** Toward international standardization of superconducting materials; development of standard measurement methods for critical current and a.c. losses *Cryogenics* (1992) **32** 557

9 **Kramer, E.J.** Scaling laws for flux pinning in hard superconductors *J Appl Phys* (1973) **44** 1360

10 **Walters, C.R.** An analysis of the Nb_3Sn critical current data obtained in VAMAS round robin *Adv Cryog Eng* (1994) **40** 839

11 **Walters, C.R., Davidson, I.M. and Tuck, G.E.** Long sample high sensitivity critical current measurements under strain *Cryogenics* (1986) **26** 406

12 Certificate of Standard Reference Material 1457, Office of Standard Reference Material, NIST, Gaithersburg, MD, USA (1984)

13 **Ekin, J.W.** Strain scaling law for flux pinning in practical superconductors. Part 1: Basic relationship and application to Nb_3Sn conductors *Cryogenics* (1980) **20** 611